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Westinghouse

ELECTRIC CORPORATION

WESTINGHOUSE ELECTRIC CORPORATION

32 NORTH MAIN STREET

DAYTON 2, OHIO

INTERIM TECHNICAL PROGRESS REPORT NO. 5

500°C SILICON CARBIDE RECTIFIER PROGRAM

Covering Period 1 Oct. to 31 Dec. 1962

CONTRACT NO. AF 33(657)-7027

ASD TR 7-727(5)

ASD INTERIM REPORT 7-727(5)

January 1963

$500^{\circ}C$ Silicon Carbide Rectifier Program

H. C. Chang
R. B. Campbell
J. Ostroski
D. Barrett

Westinghouse Research Laboratories

Beulah Road, Churchill Boro

Pittsburgh 35, Pennsylvania

Contract: AF 33(657)-7027

ASD Project 7-727

Interim Technical Progress Report No. 5

1 October 1962 - 31 December 1962

Aeronautical Systems Division

United States Air Force

Wright-Patterson Air Force Base, Ohio

ABSTRACT-SUMMARY

Interim Technical Progress Report ASD INTERIM REPORT 7-727(5)

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500°C Silicon Carbide Rectifier Program

H. C. Chang

et al

Westinghouse Electric Corporation

Hexagonal silicon carbide crystals are being grown in a Kroll-type furnace by the sublimation technique. In order to obtain greater control over the growth variables a new furnace has been assembled and is being used in a series of experiments. This furnace should have a higher yield than the present furnaces and should give valuable information on crystal growth parameters.

The research on grown junction crystals has emphasized the preparation of crystals with a high degree of surface and internal perfection by studying the effect of radiation sinks and cavity geometry.

The methods and materials of device fabrication are being studied and adapted to meet stability and quantity processing requirements. Electrical characteristics at various temperatures are given for several encapsulated rectifiers. The electrical properties of these rectifiers had been stabilized by an ambient atmosphere treatment prior to encapsulation.

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NOTICES

FOREWORD

This Interim Technical Progress Report covers work performed under contract AF 33(657)-7027 from 1 September 1962 to 31 December 1962. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with Semiconductor Department, Westinghouse Electric Corporation, Youngwood, Pennsylvania was initiated under ASD Project 7-727, "500°C Power Rectifier."

Dr. H. C. Chang, Advisory Physicist, Semiconductor Department, Westinghouse Research Laboratories was the Principal Investigator of Phase I of this project. Others who cooperated in the research and in the preparation of the report were: Dr. R. B. Campbell, Senior Physicist, Mr. J. Ostroski, Senior Chemist; and Mr. D. Barrett, Research Chemist; all members of the Power Device Department of the Westinghouse Research Laboratories.

Your comments are solicited on the potential utilization of the information contained therein as applied to your present or future production programs.

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I. INTRODUCTION

The major work of this project involves the crystal growth of silicon carbide and device fabrication. The sublimation method of crystal growth has been emphasized and certain refinements have been made on this technique. At present, only this method has produced crystals suitable for the fabrication of 1 amp - 150 volt rectifiers. However, in view of the inherent difficulties in this, several other vapor phase growth methods are being examined.

The rectifier fabrication study has emphasized the techniques for stabilizing the electrical characteristics at elevated temperatures, the evaluation of component parts used for encapsulating the crystals and the refinement of techniques for quantity fabrication.

Since the major limiting factors in the sublimation method of crystal growth appear to be an uncontrolled nucleation reaction and an insufficient control of growth temperature, certain refinements of the sublimation technique are being investigated with a newly designed sublimation furnace. Present investigation includes the effect of substrate and radiation sink size and placement on crystal nucleation and growth. Experiments are also being made to increase the yield of usable crystals in the sublimation furnace used for preparing grown junctions by the use of larger cavities and various radiation sinks.

The devices that have been fabricated during this program have been prepared from crystals grown by the sublimation method. Important progress has been made in reducing and stabilizing the reverse current at elevated temperatures with the use of ambient atmospheres. Ambient atmospheres that have been studied include, helium, water-vapor, argon, and argon-hydrogen. The best results have been obtained with helium and water vapor and these are being used in processing and encapsulating the rectifiers. Most of the treated rectifiers have shown more stable electrical characteristics at elevated temperatures than those not treated. In this period eight rectifiers have been treated in helium or water vapor and several of them are discussed in detail. Further studies have been made of the components used for encapsulation, and the maximum temperature at which these components are usable has been increased.

II. Crystal Growth

1. Newly Designed Sublimation Furnace

The detailed features of this furnace and the results of the initial runs have been discussed in earlier reports.

After a number of runs it became obvious that the graphite parts which initially had been designed were not suitable for making the necessary study of the effect of cavity geometry and size on the crystal growth parameters. The substrate (thin inner graphite cylinder on which the silicon carbide crystals nucleate) generally used is 1-1/4 inches in diameter. The reasons for investigating larger substrates can be seen as follows. The crystal growth occurs by deposition of the silicon and carbon vapor species on the growing surfaces of the crystal. To continue growth, the crystal surfaces must then radiate heat to some cooler region. These cooler regions are generally provided by radiation heat sinks attached to the end caps of the substrate. With a small-sized cavity, there is less chance to control this radiation loss and therefore the growing crystals may be small and imperfect. With a larger sized cavity, the growth should proceed at a more controllable rate and larger, more uniform crystals should result.

Newly designed internal graphite parts were fabricated and installed in the furnace. These parts permit use of a substrate cavity up to 3 inches in diameter.

A series of runs have been made using a 3 inch diameter substrate. Since the crucible size is the same, the amount of raw material that can be used is less due to the large volume of the cavity. It has been found that the growth conditions (temperature, temperature gradient, etc.) are not as easily controlled as with the smaller sized substrate. The crystals grown thus far have not been as large as expected, and in several runs only a very small number of nucleation sites were noted on the inner wall. It is believed that this lack of crystal growth is primarily due to inefficient radiation from the growing edge. More efficient radiation sinks are being designed and fabricated to test this hypothesis. While this is being done, runs are continuing using 1-1/2 and 2 inch diameter substrates. These runs have shown no great improvement over the first runs using a 1-1/4" dia. substrate. Also planned for the near future are runs using a very small substrate (about 1/2" diameter). The effect of substrate size has been investigated previously, but the interrelation between the size of the cavity and the radiation sinks may well give valuable information on the crystal growth.

2. Initial Style Sublimation Furnace

A series of experiments have been carried out in this furnace to determine the effect of a hydrogen ambient atmosphere on the crystal purity and size. The hydrogen concentration in argon was 5%, 10%, and 90%. In addition a series of runs were made in pure hydrogen.

The results of runs using the lower concentrations of hydrogen (5 and 10%) were not noticably different than those made in pure argon. In using the larger concentrations of hydrogen the most difficult problem is obtaining the proper temperature configuration in the charge. This is much more difficult with hydrogen than with argon due to the ten-fold greater thermal conductivity of hydrogen. This problem was never completely solved, but the addition of extra powdered insulating material around the growth cavity was of value. Both the grown crystals and the charge material produced in these runs were white, showing none of the green coloration noted in the runs using the argon ambient. Several of these crystals were tested using the method of Exciton Spectroscopy (cf W. J. Choyke and Lyle Patrick, Phys. Rev. 127 1868, 1962). No quantitative results are available, but the concentration of nitrogen is low. Resistivity and Hall measurements will be made on these crystals.

The crystals prepared thus far are not of device quality mainly due to improper growth conditions. These conditions cause the crystals to be stepped, contain inclusions, etc.

One disadvantage of growing the silicon carbide crystals in a hydrogen ambient is the reaction of hydrogen with carbon at elevated temperatures. It has been shown that hydrogen will transport carbon at elevated temperatures by the formation of acetylene. Therefore, the usable life of the internal graphite parts is shortened by the use of hydrogen.

The results of these growth experiments using hydrogen are not completely unexpected. Some early work on another project briefly studied the effect of the hydrogen ambient and found an apparent increase in crystal purity. These experiments were not continued at that time due to the thermal conductivity problems.

3. Grown Junction Crystals

The preparation of grown junction crystals is continuing on a normal basis.

Single crystals of silicon carbide containing p-n junctions were grown by the sublimation technique. Crystals from the various runs have been selected and fabricated into 1 ampere rectifiers.

As has been described in previous reports, the growth process consists of subliming an aluminum doped charge of silicon carbide and condensing the vapor as single crystal platelets. Nitrogen gas is admitted slowly into the furnace during the latter stages of growth in amount sufficient to compensate the aluminum acceptor density with a nitrogen donor density. As the nitrogen partial pressure is increased, the growing crystals acquire a majority of donor impurities. In this manner graded junctions are formed.

Usually, only a small percentage of these crystals can be processed into 1 ampere diodes due to (1) improperly formed p-n junction region, and (2) imperfect crystal structure. The p-n junction electrical properties are a function of the rate of increase in nitrogen partial pressure during growth. While the rate of increase can be held constant, the individual crystals grow at different rates; therefore, the junction impurity gradients and depletion widths will vary. Only a few of the crystals in a given run will have the correct compensated impurity gradient. The measurement of impurity gradients and junction depletion widths have been described in previous reports.

The crystalline imperfections and differing growth rates of the crystals are mainly due to interference during growth by closely neighboring crystals. The crystals nucleate at random on the cavity substrate and there is insufficient condensation of vapor for the growth of crystals which are close together. Due to their proximity, the radiation from the growing surfaces is limited, and intergrowth, imperfections, and differing growth rates result. Well formed platelets in the cavity are only found separated from neighboring crystals where growth conditions are more nearly uniform.

Experiments undertaken in the grown junction technique involve, (1) control of the number and geometric spacing of nucleations on the cavity substrate, (2) uniformity of thermal gradients about the cavity, (3) providing a radiation sink which can be uniformly "seen" by all growing platelets. In this quarter experiments were made to increase the yield of usable crystals using a larger cavity and a gas tube radiation sink. Twenty preparations were made, eleven for p-n junction crystals and nine for p-type substrates for epitaxial deposition. Four of the p-type preparations were made while adjusting thermal conditions after the furnace heating element and heat shields had been replaced.

The cavity within the charge was increased in diameter from 1.25 to 2.0 inches. This larger condensation space resulted in growth of some larger crystals with slightly higher perfection. Since the degree of reproducibility is low in the sublimation growth technique, more experiments must be made to verify this effect. The gas tube radiation sink consisted of a 1/4-inch diameter graphite tube which passed axially through the cylindrical cavity. A regulated flow of argon gas was passed through the tube to control the amount of heat dissipation. With low gas flow rates the tube clogged with silicon carbide deposits, but remained clear at high flow rates. The experiments were not uniformly successful, but indicated that a differently shaped sink with the gas flow would be advantageous. The radiation sink will be shaped to offer a larger solid angle through which the growing crystals could radiate excess energy.

In another experiment, crystals were grown in a cavity using a cone shaped radiation sink which extended from the cavity up above the charge to dissipate heat more effectively. An identical preparation was made next without the heat dissipator but with a cone shaped sink. The crystals were larger when the dissipator was used.

III. Device Fabrication

1. Introduction

During this period the work on the device fabrication phase of the project has emphasized a continuing investigation into the beneficial effects of exposing rectifier units at 500°C to various gases, and further testing of encapsulation components. The ambient atmosphere effect has been described in detail in the fourth quarterly report of this contract. The behavior of further representative units is reported here.

2. Electrical Characteristics

Due to the results reported earlier, all silicon carbide diodes which are to be encapsulated as rectifiers are exposed to a gas at elevated temperatures after they have been provided with bases and contacts. This treatment consists of heating the rectifier to 500°C in vacuo with no forward current but a peak reverse voltage of 150v PRV. After initial electrical measurements at this temperature, a gas (generally helium or water vapor) is admitted and the change in the electrical characteristic noted.

Eight rectifiers have been treated in helium and water vapor as described. After treatment they were cleaned ultrasonically and a header welded to the base. To further test the ambient effect, some were evacuated to 2×10^{-5} mm Hg and sealed, while others were evacuated, backfilled with one half atmosphere of helium and then sealed. At this time no significant difference between the two methods of encapsulation have been noted. In the following, data for five representative rectifiers is given.

1. Rectifier L-35 was processed using helium as the ambient gas. The initial half wave average reverse current at 150 volts PRV and 500°C ambient was 4.0 mA. After 10 minutes in helium the reverse current decreased to 92 μA and remained stable at this current. The stability of the reverse current was tested several days later at 150 volts PRV and 500°C ambient and no increase was noted. The results of the test are shown in Figure 1.

2. Rectifier L-34 was processed using water vapor as the ambient. The initial half wave average reverse current at 150 volts PRV and 500°C ambient was 165 μA. After 10 minutes in water vapor the reverse current decreased to 80 μA. The results of this test are shown in Figure 2. After several hours at 500°C case temperature the rectifier was subjected to a peak reverse voltage of 275 volts and a case temperature. The rectifier showed a reverse current of 330 μA at these conditions. The stability at this temperature was very good. The results of this test at 510°C case temperature and room temperature are given in Figure 3.

After ultrasonic cleaning in alcohol, the rectifier was welded to a gold-bonded ceramic to stainless steel header. After evacuating to a pressure of 2×10^{-5}mm Hg the unit was sealed and tested at a forward current of 1 ampere and peak reverse voltages to 200 volts at various temperatures to 500°C. The results are shown in Figure 4. The reverse current of 550 μA is fair at 500°C considering that the forward voltage of this rectifier is relatively high. This high forward voltage contributes to the raising the junction temperature above the normal value thus giving a higher reverse current. The average forward voltages at different temperatures of this unit are given in Figure 5.

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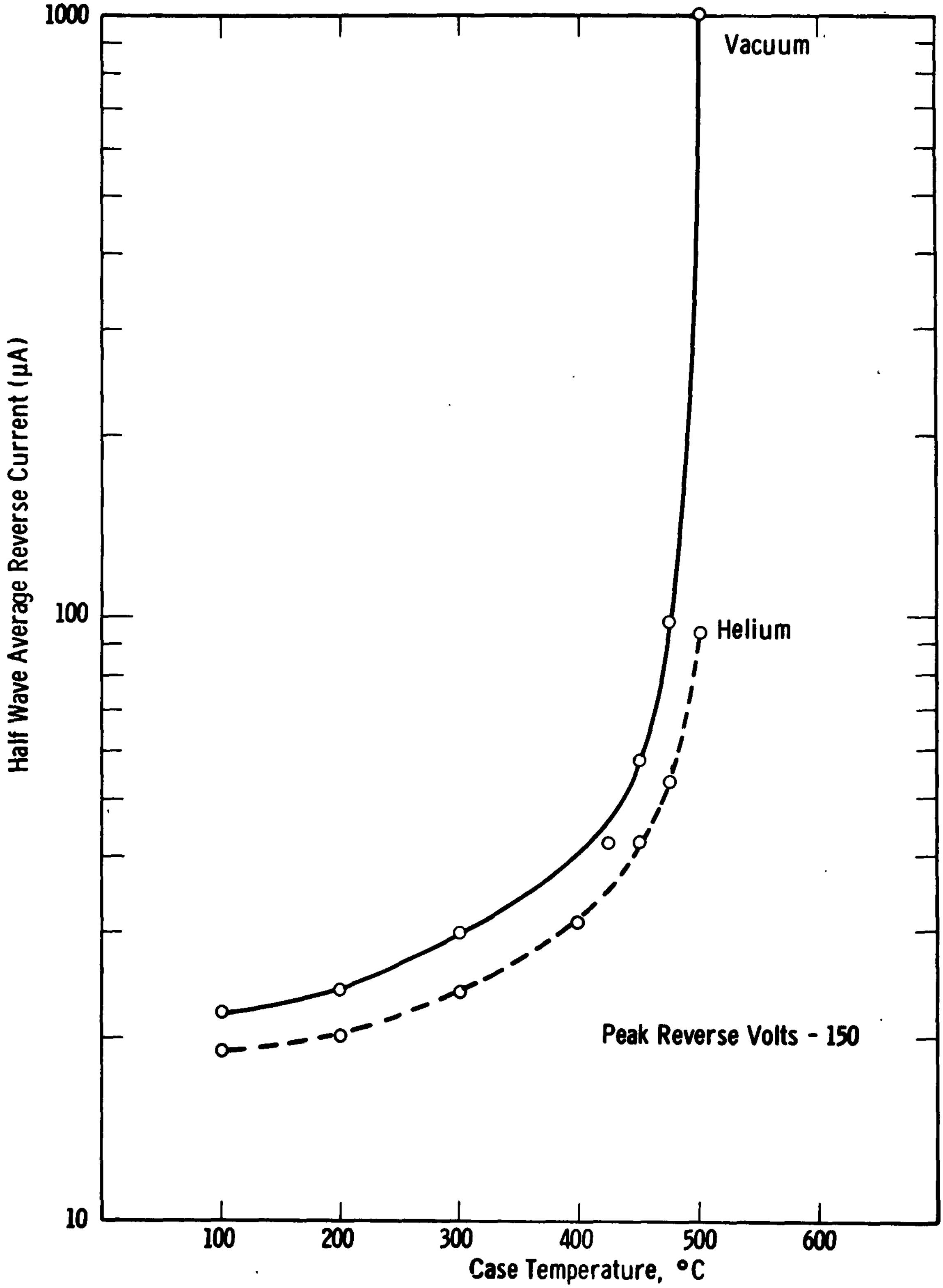

Fig. 1

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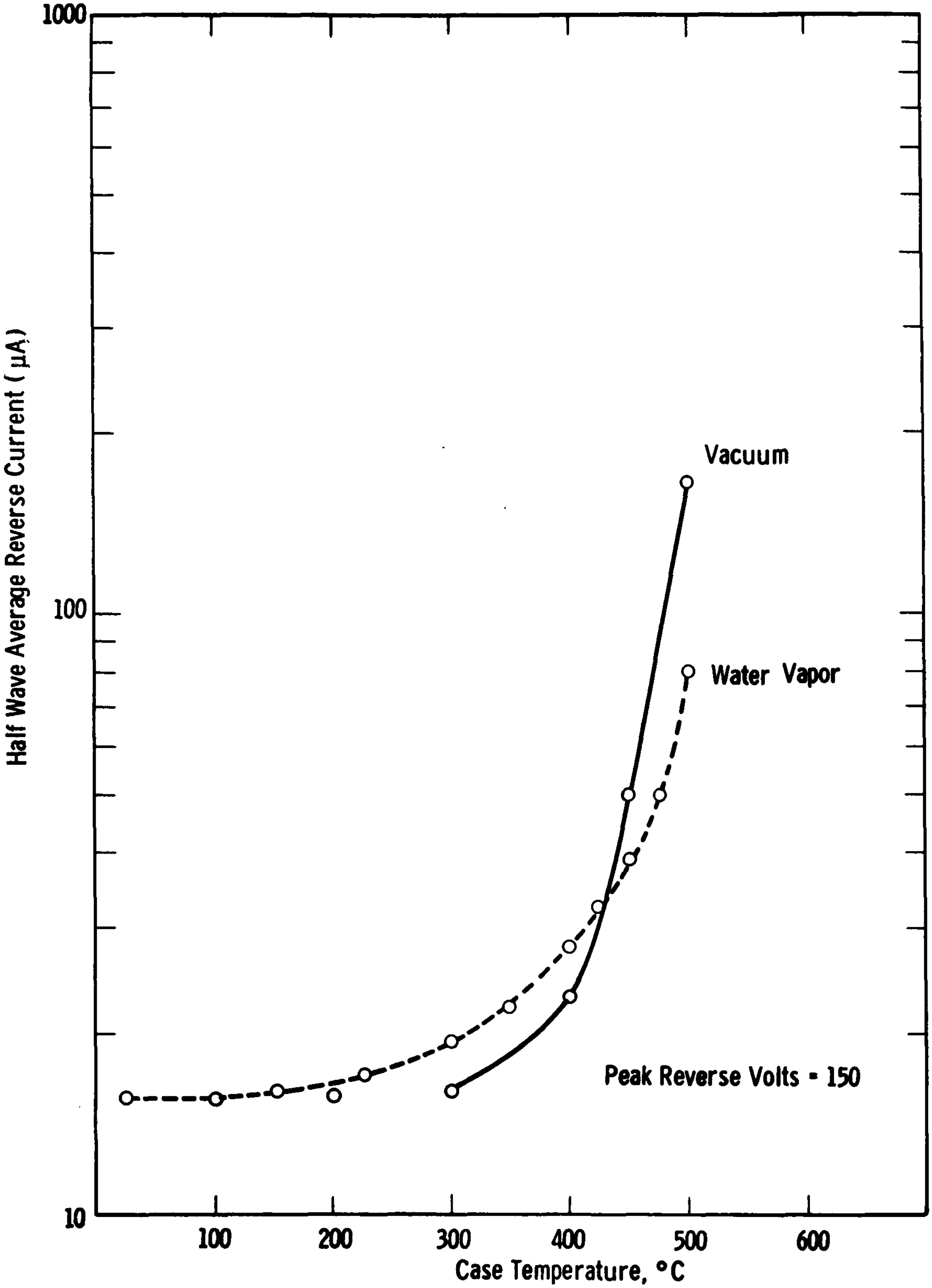

Fig. 2

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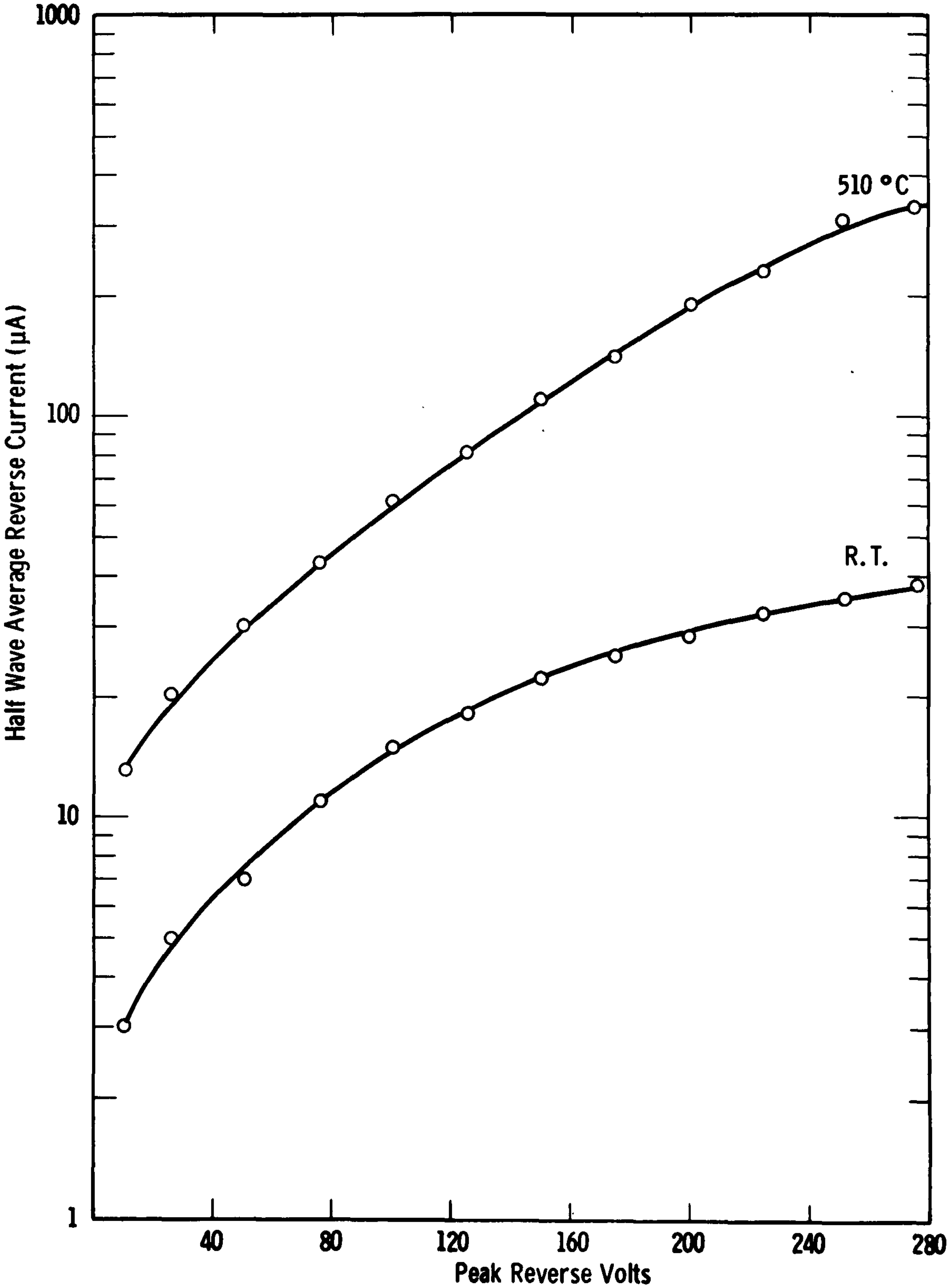

Fig. 3

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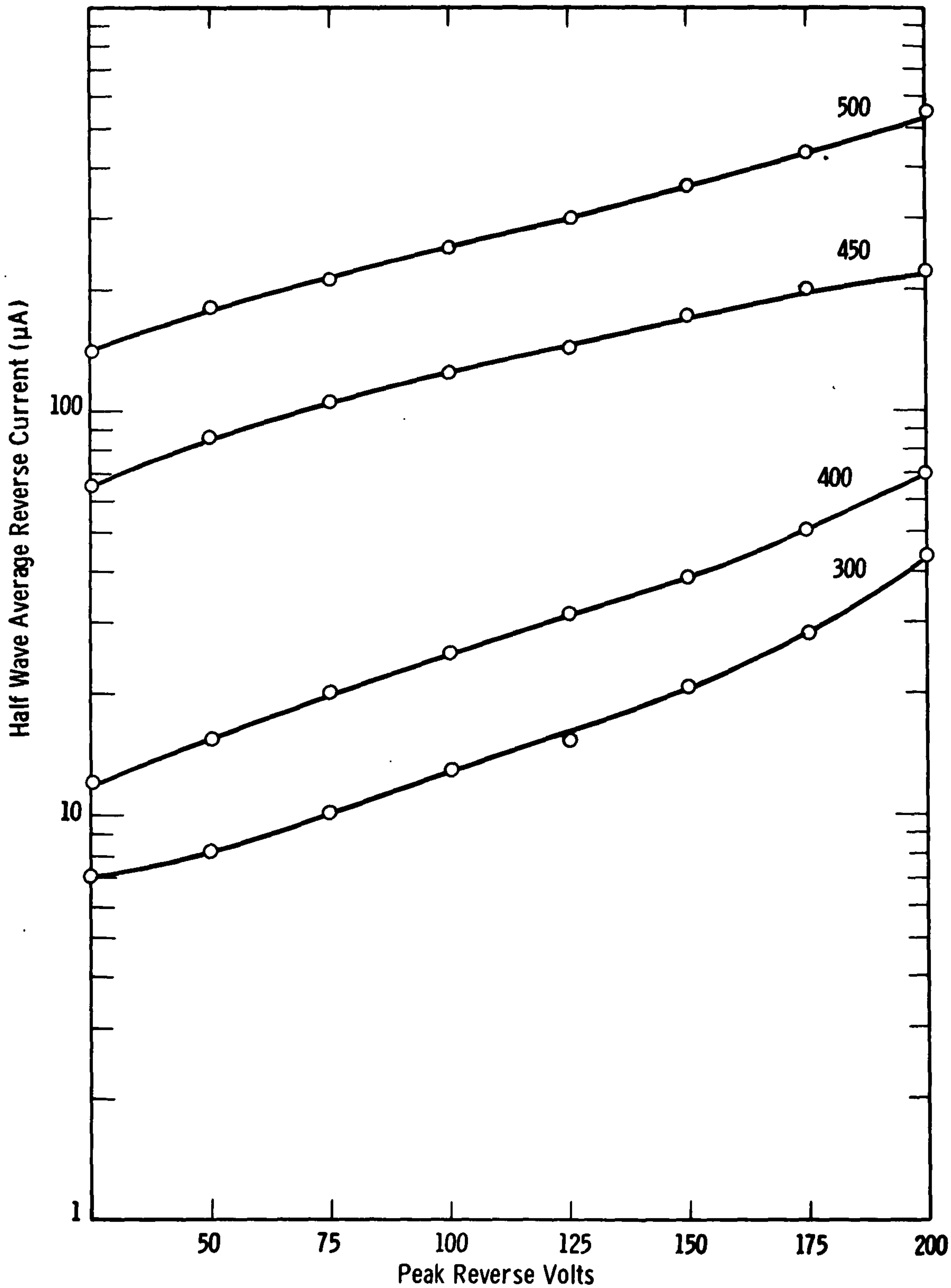

Fig. 4—Half wave average reverse current vs. peak reverse volts at average forward current = 1 ampere-case temperature = °C

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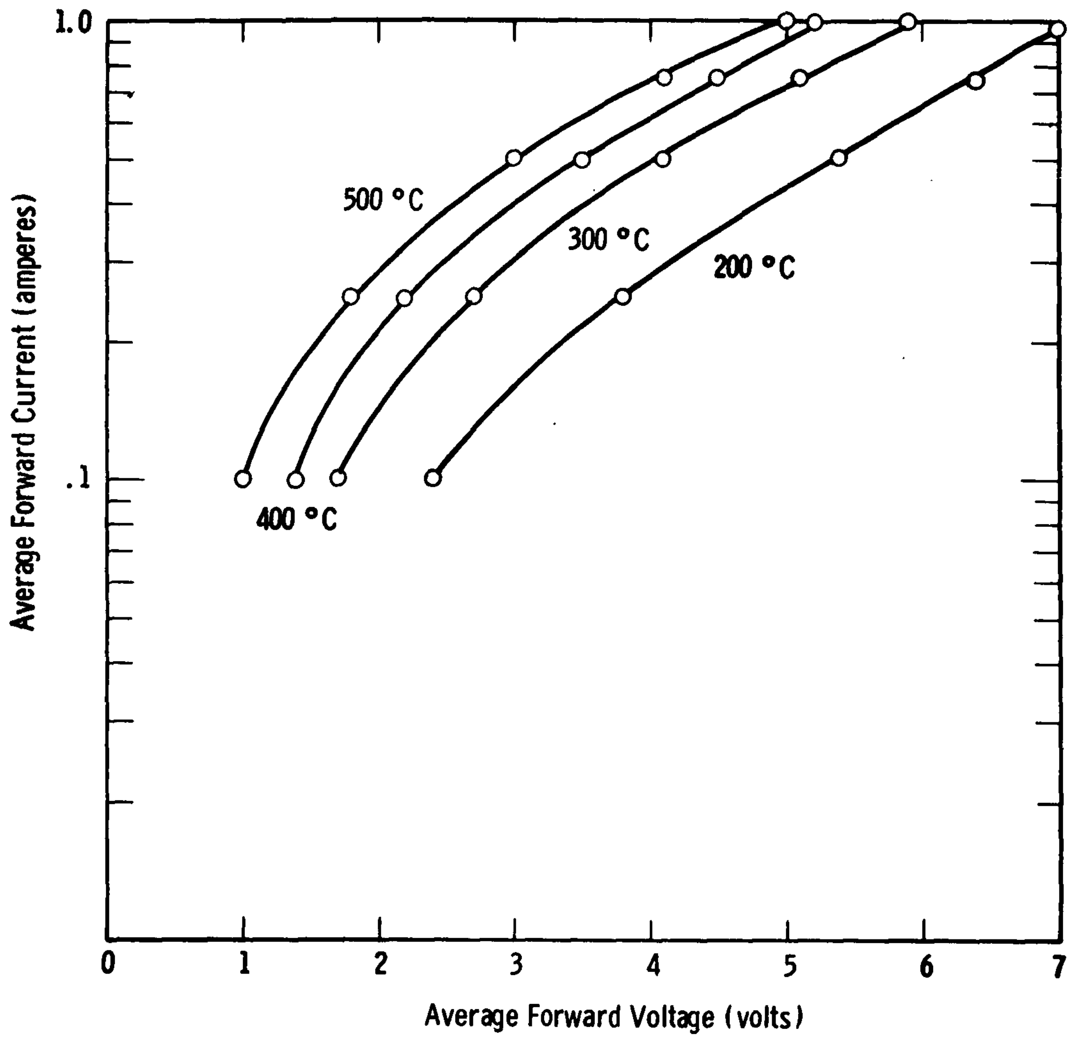

Fig. 5—Average forward current vs. average forward voltage-case temperature °C peak reverse volts 150

3. Rectifier L-33 was treated in helium. It showed an initial low forward voltage and a low reverse current. Past experience has indicated that under these conditions, treatment with helium or water vapor would reduce the reverse current by a small amount. But this small amount would be significant at 500°C and with a forward current. The reverse current on this unit was reduced from 67 μA at 150 volts PRV and 500°C to 45 μA. The rectifier was then encapsulated and the electrical characteristics were determined. Figure 6 and 7 show the forward and reverse characteristics of the rectifier after treatment with helium and encapsulation. The average forward voltage at 1.25 amperes forward current, 150 volts PRV and 500°C case temperature showed 3.25 volts while the half wave average reverse current showed 155 μA at a peak reverse voltage of 200 volts and a 1 ampere forward current.

These results and those on other encapsulated units have verified our initial results on this method. There is no doubt of its beneficial action. Experiments designed to explain this passivation mechanism are now underway, but as yet no definite conclusion can be given.

3. <u>Encapsulation</u>

To obtain further data on the gold-bonded ceramic to stainless steel header it was necessary to extend the testing temperature to 650°C. This is about 50°C above the storage temperature requirement.

A rectifier L-31 was processed as usual with gold-tantalum and tungsten backing plates and brazed to a nickel base. The regular helium treatment was omitted. The header was welded to the base, evacuated,

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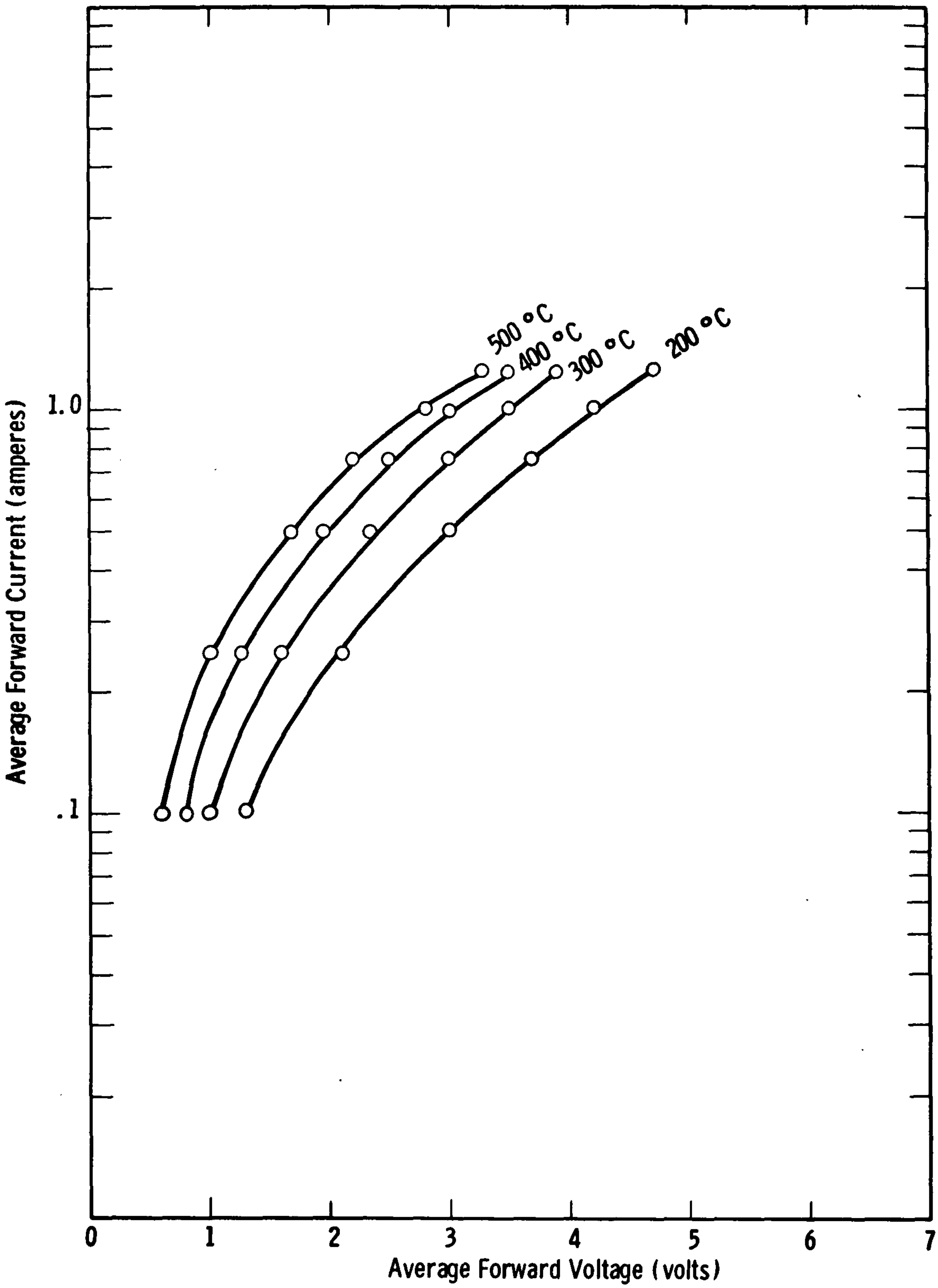

Fig. 6—Average forward current vs. average forward voltage and case temperature °C 150 volts PRV.

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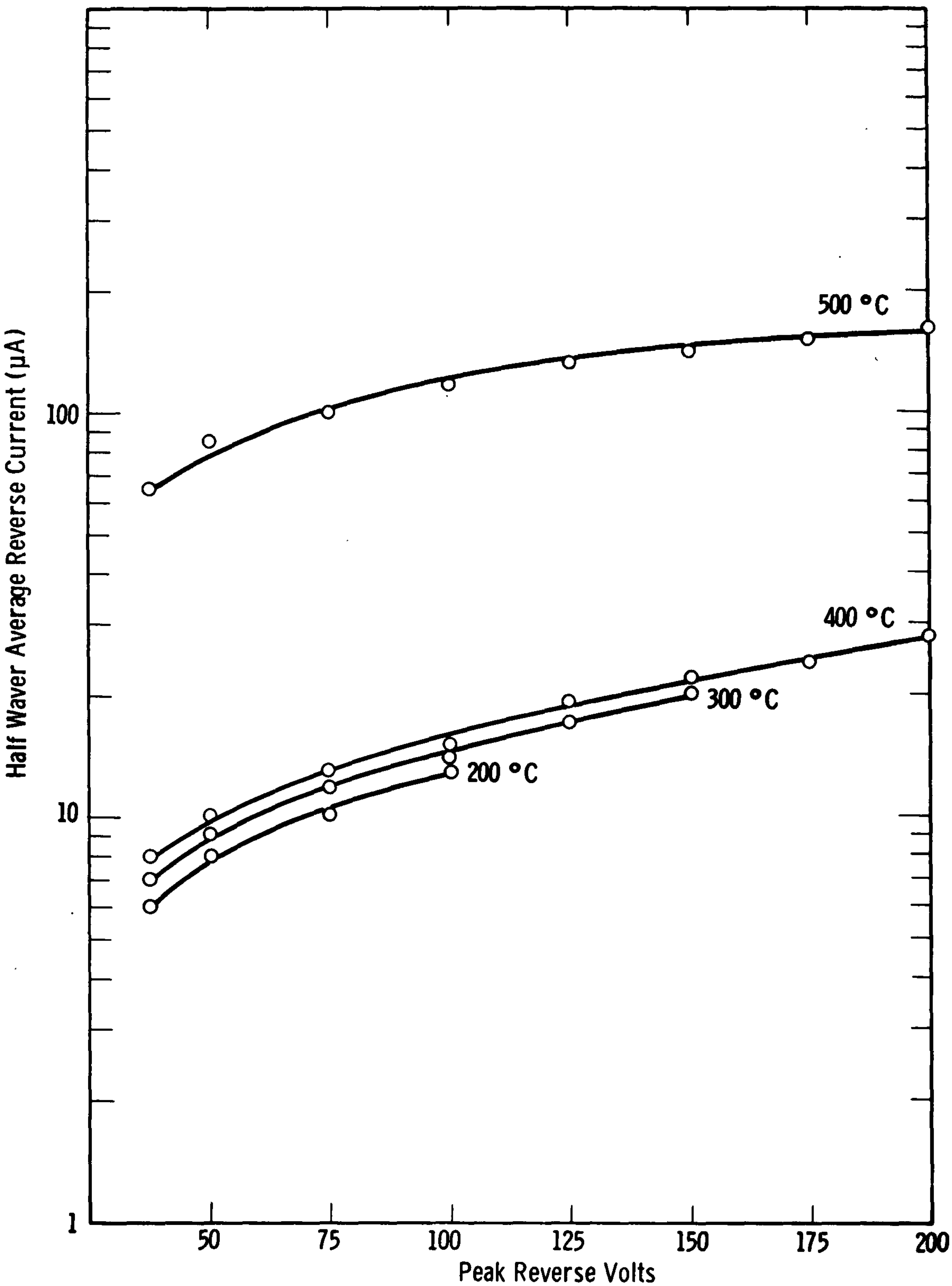

Fig. 7—Half wave average reverse current vs. peak reverse volts at average forward current = 1 ampere-case temperature °C

flushed several times with helium and finally backfilled with helium to half an atmosphere. After sealing the rectifier was placed in an oven. The temperature was raised to 650° and maintained between 600° and 650°C for at least 1 hour. No significant change in the reverse current at 150 volts PRV was noted. Figure 8 shows the results of this test where a maximum reverse current of 2.0 mA was reached at 650°C and 150 volts PRV. Microscopic examination of the header did not show any excess corrosion of the vital joints.

Another rectifier L-35A which had been treated with helium before encapsulation was used to obtain an approximation of the junction temperature at 500°C under a load current. A half wave average reverse current of 760 μA was obtained under a 1 ampere forward current at 150 volts PRV and 500°C case temperature. The rectifier was then tested under a static no load condition by raising the temperature until the same reverse current (760 μA) was obtained.

This temperature was 575°C, thus it appears that with a case temperature of 500°C and a forward current of 1A, the junction temperature rises 75°C to 575°. This is shown in Figure 9.

The initial test results on the silver-nickel bases have been analyzed but are inconclusive. Further work will be required before an evaluation can be made.

IV. Conclusions and Future Plans

Experiments with the new furnace and newly designed internal graphite parts should give valuable information on crystal growth.

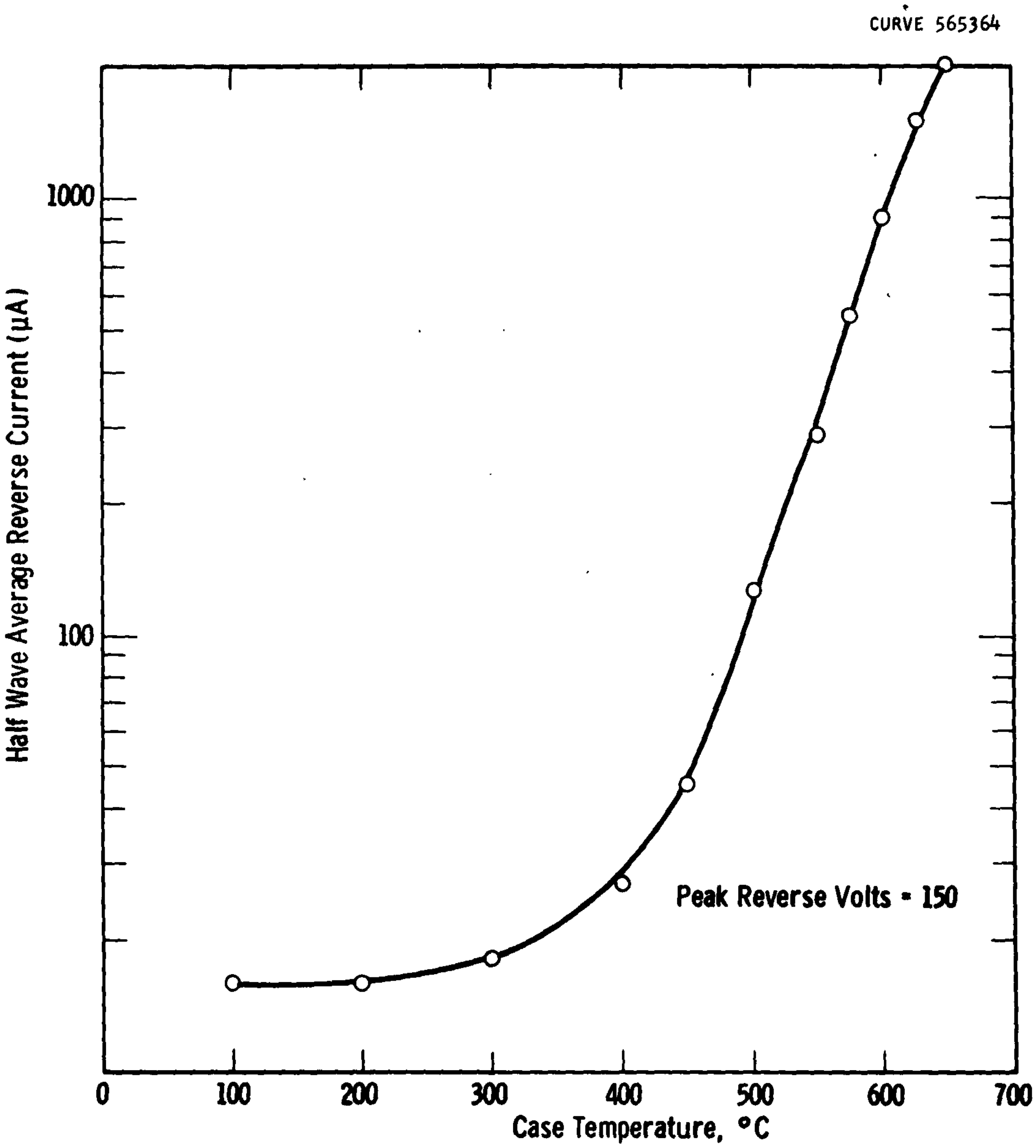
CURVE 565364
Half Wave Average Reverse Current (μA)
1000
100
Peak Reverse Volts = 150
0
100
200
300
400
500
600
700
Case Temperature, °C

Fig. 8

CURVE 565365

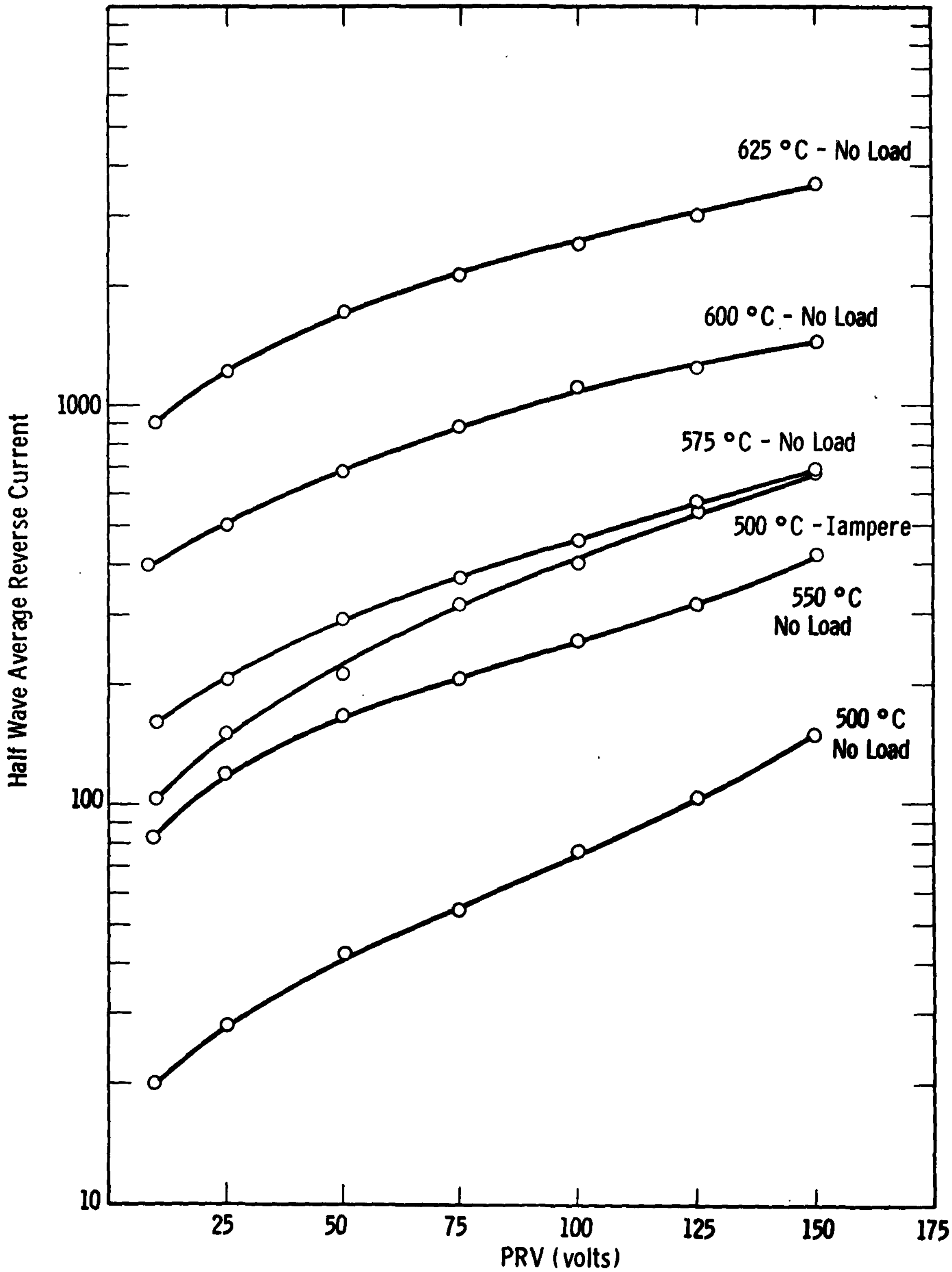

Fig. 9

Efforts in grown junction crystal growth will be extended in the area of radiation sinks and cavity geometry to increase the yield of usable crystals.

Effects of various ambient atmospheres on silicon carbide rectifiers have been adapted for encapsulating rectifiers.

During the next period we plan to accomplish:

1. Continue experiments with the new furnace.

2. Continue the preparation of grown junction crystals suitable for fabrication of 1 ampere rectifiers.

3. Investigate the effect of shaped radiation sinks and cavity geometry on the yield of more usable crystals.

4. Results from the ambient testing will continue to be adapted to encapsulating silicon carbide rectifiers.

5. Continue the encapsulation of rectifiers to obtain further electrical data.

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